REMARKS

Applicants respectfully request reconsideration and allowance of the pending claims.

I. Status of the Claims

Claims 48, 49, 52, 53, 59, 61, and 66 remain pending, and claims 67, 68, and 69 have been added.

Claim 48 has been amended to clarify that the nickel layer is deposited with an internal compressive stress at the high plating rate throughout the throughput of 14 to 22 MTO, which is supported at paragraphs [0020] and [0036]. Claim 48 has also been amended to require metal salt stabilizers comprising volatile anions, which is supported at paragraph [0017].

New claim 67 is supported at paragraph [0017]. New claims 68 and 69 are supported at paragraphs [0051] and [0052].

II. Claim Rejections under 35 U.S.C. §103(a)

A. Over GB 1,243,134

Reconsideration is requested of the rejection of claims 48, 49, 52, 53, 59, 61, and 66 as being obvious over GB 1,243,134 in view of Martyak et al. (U.S. 5,258,061) and Zhong et al. (U.S. 6,106,927).

1. The Claimed Invention

Claim 1 is directed to a method for electroless deposition of a nickel layer with internal compressive stress on a substrate, the method comprising:

> contacting the substrate with an electrolyte comprising a nickel metal base salt, a reducing agent, a complexing agent, an accelerator, and a stabilizer to thereby electrolessly deposit the nickel layer with

> internal compressive stress on the substrate, wherein the nickel layer with internal compressive stress is electrolessly deposited at a deposition rate of 7 µm/h to 14 µm/h with a throughput of 14 to 22 MTO;
>
> wherein the nickel metal base salt of the electrolyte comprises an anion selected from the group consisting of acetate, formate, nitrate, oxalate, propionate, citrate, ascorbate, and combinations thereof; ...
>
> wherein the complexing agent is selected from the group consisting of 2-hydroxypropanoic acid, propanedioic acid, and a combination thereof; ...
>
> wherein the stabilizer is a metal salt wherein the metal is selected from the group consisting of lead, bismuth, zinc, tin, and combinations thereof and the metal salt comprises an anion selected from the group consisting of acetate, formate, nitrate, oxalate, propionate, citrate, ascorbate, and combinations thereof; and ...

Applicants' method as defined by claim 1 is directed to the electroless deposition of nickel layers having internal compressive stress. The method is accomplished by employing an electrolyte comprising metal sources such as the nickel metal base salts and the metal salt stabilizers which comprise volatile anions and by the selection of complexing agents. It is critical to note that applicants' claimed method requires not just that volatile anions be present in the electrolytic solution, but that the metal sources themselves (both nickel ions and metal stabilizers) comprise volatile anions. This aspect of applicants' claim cannot be ignored when assessing the patentability of the claim. By selecting sources of nickel deposition ions and metal stabilizers which have volatile anions, the plating speed may be maintained within the desired interval (i.e., between 7 µm/h to 14 µm/h) during an extended useful life of the electrolyte (i.e., 14 to 22 MTO).

Moreover, claim 48 not only requires that the useful life of the bath be extended to between 14 and 22 MTO but that the nickel layer is deposited with internal compressive stress throughout the extended useful life of the electrolyte. That is, the nickel layer has internal compressive stress initially, at 1 MTO, at 2 MTO, at 3 MTO, and so on through at least 14 MTO, 15 MTO, 16 MTO, 17 MTO, or more, up to 22 MTO. See paragraphs [0020] and [0036].

A nickel layer having internal compressive stress is characterized by a stress that results in internal compaction (decrease in volume), thereby leading to a nickel layer that is pore-free and crack-free. See applicants' specification at paragraph [0020]. This is in sharp contrast to a metal layer having tensile stress, which is a force that causes elongation, or an increase in volume. As further shown in the attached Declaration by Inventor Franz-Joseph Stark and further described herein, not all metal layers necessarily have a compressive stress. Applicants' inventive method therefore provides a method for depositing a nickel layer having internal compressive stress over an extended useful life of the electrolyte.

2. Disclosures of the prior art

GB1,243,134 discloses a method for electroless deposition of nickel-phosphorus layers from an electroless deposition composition comprising nickel ions, glutamic acid or a glutamate salt is used as a complexing agent, and a source of hypophosphite as a reducing agent. See page 2, lines 9-24 and 46-56. While page 2, lines 90-94 of the reference lists several sources of nickel including nickel sulfate hexahydrate, nickel chloride, nickel bromide, or nickel acetate, each example uses nickel sulfate hexahydrate. Optional materials include succinic acid and thiocyanate exaltants. See page 2, lines 46-61.

The GB1,243,134 reference is entirely silent on the stress properties of their nickel layers, does not disclose that the nickel layer has compressive stress at any stage during the deposition, and does not disclose that the compressive stress of the deposited nickel is maintained through 14 to 22 MTO. Moreover, GB1,243,134 does not disclose any of the metal salt stabilizers required by claim 1, nor does the reference place any importance on selecting metal sources comprising volatile anions to enhance not only the useful life of the electrolyte but to also affect the stress properties of the nickel layer such that the nickel layer has compressive stress through 14 to 22 MTO.

Martyak et al. (U.S. 5,258,061) disclose a method for recycling electroless nickel plating solutions using solvent extraction and anion filtration. Martyak et al. discuss a variety of nickel salts at Col. 2, lines 13-21 and a variety of chelating agents at Col. 2, line 21-31. Stabilizers are discussed at Col. 2, starting at line 59, including amines such guanidine, dimethylamine, and others and acids such as taurine, 2-hydroxyethanesulfonic acid, and others.

Martyak et al. do not disclose the stress properties of their nickel layers and do not disclose that the nickel layer has compressive stress and that the compressive stress is maintained throughout the useful life of the electrolyte. Moreover, Martyak et al. do not disclose any of the metal salt stabilizers required by claim 1, nor do they place any importance on selecting metal sources comprising volatile anions to enhance not only the useful life of the electrolyte but to also affect the stress properties of the nickel layer.

Zhong et al. (U.S. 6,106,927) disclose a method for depositing ultra-smooth nickel-phosphorus layers by an electroless deposition method. This may be accomplished by incorporating metal ions, such as aluminum ions and copper ions, into the electroless plating bath. Zhong et al. discuss potentially applicable complexing agents at Col. 5, lines 10-16. Zhong et al. cite several stabilizers including lead, antimony, mercury, tin, and iodate at Col. 5, lines 17-24, yet they place no particular importance on the source of these stabilizers and provide the ordinarily skilled person with no basis for appreciating that volatile anion sources of these stabilizers and volatile anion sources of the nickel ion enhance either the stability of the electrolyte or affect the stress properties of the deposited nickel layer. In fact, Zhong et al., like the other references cited, do not disclose anything related to the stress properties of their deposited nickel layers.

3. The Cited References Fail to Support a *Prima Facie* Case of Obviousness

The Office's *prima facie* case of obviousness is deficient since the cited references do not disclose an electroless deposition method employing an electrolyte that has an extended useful life that further deposits a nickel layer having internal compressive stress throughout 14 to 22 MTO.

In the context of determining obviousness under 35 U.S.C. §103(a), "The Supreme Court in *KSR* reaffirmed the familiar framework for determining obviousness as set forth in *Graham v. John Deere Co*. (383 U.S. 1, 148 USPQ 459 (1966)),..." See MPEP §2141. Within that framework, the Office must determine obviousness based upon underlying factual inquiries, including:

(A) Determining the scope and contents of the prior art and
(B) Ascertaining the differences between the prior art and the claims in issue.

With regard to the electroless processes described in the cited references, GB1,243,134 discloses a method wherein the base composition may comprises a source of nickel ions selected from among a variety of salts, a source of glutamate as a complexing agent, and a source of hypophosphite as a reducing agent. Martyak et al.'s composition may comprise a variety of nickel salts, chelating agents, and stabilizers including amines such guanidine, dimethylamine, and others and acids such as taurine, 2-hydroxyethanesulfonic acid, and others. Zhong et al., likewise, disclose a wide selection of nickel salts, chelating agents, reducing agents, and stabilizers.

A critical *Graham* difference between the cited references and applicants' claims is that applicants select only those nickel salts and metal stabilizers salts comprising volatile anions since to enhance both the stability of the electrolyte and ensure that the deposited nickel layer is characterized by internal compressive stress and such compressive stress is a characteristic of the nickel layer for the entire extended throughput, while the cited references are entirely silent on the stress characteristics of the nickel layer and do not provide the ordinarily skilled person with any reason to carefully select the metal sources used in preparing their electrolytes, for the purpose of depositing nickel layers having internal compressive stress or for any other reason.

The provision of a clearly articulated reason why the ordinarily skilled person would have modified the disclosure of the cited reference in order to reach applicant's claimed method is needed in establishing the *prima facie* case under the relevant case law and the MPEP guidelines as shown at §2142:

> The key to supporting any rejection under 35 U.S.C. **103** is **the clear articulation of the reason(s)** why the claimed invention would have been obvious. The Supreme Court in *KSR International Co. v. Teleflex Inc.*, 550 U.S. ___, ___, 82 USPQ2d 1385, 1396 (2007) noted that the analysis supporting a rejection under 35 U.S.C. **103** should be made explicit. The Federal Circuit has stated that "rejections on obviousness cannot be sustained with mere conclusory statements; instead, **there must be some articulated reasoning with some rational underpinning** to support the legal conclusion of obviousness." *In re Kahn*, 441 F.3d 977, 988, 78 USPQ2d 1329, 1336 (Fed. Cir. 2006). See also *KSR,* 550 U.S. at ___ , 82 USPQ2d at 1396 (quoting Federal Circuit statement with approval). (Emphasis added)

Since the cited references place no apparent significance on the selection of the anions of their nickel sources and other materials, the ordinarily skilled person would not have discerned any reason for carefully selecting nickel and stabilizer metal salts comprising volatile anions to affect the stress characteristics of their deposited nickel layers or for any other reason.

Accordingly, since the references are entirely silent on the stress properties of their nickel layers and since the references provide the ordinarily skilled person with no basis for selecting composition components to achieve these stress properties, the cited references do not render the method defined by claim 48 obvious.

4. *Prima Facie* Obviousness is Also Effectively Rebutted by a Showing of Unexpected Properties

Applicants' claims are further non-obvious in view of the cited references since the ordinarily skilled person would not have expected that the selection of metal salt anions in the electrolyte is a critical feature in depositing a nickel layer having internal compressive stresses for up to 22 MTO. This property is an unexpected result that effectively rebuts the *prima facie* case of obviousness. See MPEP §2145:

> Rebuttal evidence may also include evidence that the claimed invention yields unexpectedly improved properties or properties not present in the prior art. Rebuttal evidence may consist of a showing that the claimed compound possesses unexpected properties. *Dillon*, 919 F.2d at 692-93, 16 USPQ2d at 1901. A showing of unexpected results must be based on evidence, not argument or speculation. *In re Mayne*, 104 F.3d 1339, 1343-44, 41 USPQ2d 1451, 1455-56 (Fed. Cir. 1997) (conclusory statements that claimed compound possesses unusually low immune response or unexpected biological activity that is unsupported by comparative data held insufficient to overcome *prima facie* case of obviousness).

With reference now to the attached Declaration, Inventor Franz-Josef Stark prepared six electroless nickel deposition compositions. In three of the compositions (1, 3, and 5), the components were selected to include volatile anions. The other three compositions (2, 4, 6) are conventional electroless nickel deposition compositions and, as such, contain commonly used nickel sources, such as nickel sulfate hexahydrate and nickel hydroxy carbonate.

Nickel deposition from each of the three compositions of the invention was sustainable through 20 MTO (compositions 1 and 3) or 16 MTO (composition 3). Moreover, the deposited nickel layer had internal compressive stress through each and every MTO. In sharp contrast, nickel deposition from the conventional solutions was sustainable only through 8 MTO (compositions 2 and 4) or 6 MTO (composition 6). While compositions 2 and 6 may have deposited nickel layers with internal compressive stress, the effect was temporary, lasting in each case through about 3 or 4 MTO.

Based on these results, Inventor Franz-Josef Stark states:

> (12) In view of the foregoing and in view of the reproducibility of these results, I conclude that the use of volatile anions in the metal ion sources is a **critical feature** of the electroless nickel plating method of my application, since that use of volatile anions increases the useful life of the electroless nickel composition and further causes a nickel layer having internal compressive stress to be deposited throughout the composition's useful life.

Franz-Joseph Stark's Declaration therefore establishes that the use of metal sources comprising volatile anions is critical to extending the useful life of the composition and for ensuring that the nickel layers deposited therefrom have internal compressive stress throughout that useful life. Moreover, the prior art does not provide the ordinarily skilled person with any basis for appreciating the importance of carefully selecting metal salts having volatile anions for affecting the stress properties of the deposited nickel layer or for any other reason. In view thereof, applicants respectfully submit that claim 48 is patentable over the cited combination of references. Accordingly, applicants respectfully request that the rejection be withdrawn.

Claims 49, 52, 53, 59, 61, and 66 depend from claim 48 and are therefore patentable for the same reasons as claim 48 and by virtue of the additional requirements therein.

B. Over Harbulak et al.

Reconsideration is requested of the rejection of claims 48, 49, 52, 53, 59, 61, and 66 as being obvious over Harbulak et al. (U.S. 4,483,711) in view of GB 1,243,134.

The limitations and merits of claim 48 are described above in Part II.A.1. of this amendment. The disclosures of GB1,243,134 are set out in Part II.A.2. of this amendment.

Harbulak et al. disclose an electroless nickel plating method employing an electroless composition comprising a source of nickel ions, such as nickel sulfate hexahydrate, nickel chloride, and nickel acetate. See Col. 4, lines 31-38. Each composition in the Examples employed nickel sulfate hexahydrate. The reducing agent may be a source of hypophosphite, as shown at Col. 4, lines 38-44. To enhance bath longevity, complexing agents, such as lactic acid, acetic acid, succinic acid, and others may be added. See Col. 5, lines 7-20. To enhance the plating rate a sulfonium betaine may be added, such as one within the class of structures described at Col. 5, lines 27-45. Stabilizing agents such as lead, cadmium, tin, bismuth, antimony, and zinc metal cations may be added as well as cyanide and thiocyanate anions. See Col. 6, lines 10-31.

The reasons why claim 48 is patentable over the combination of Harbulak et al. and GB 1,243,134 is substantially the same as the reasons why claim 48 is patentable over the combination of GB 1,243,134; Martyak et al.; and Zhong et al.

First of all, the Harbulak et al. reference is entirely silent on the stress properties of their nickel layers, does not disclose that the nickel layer has compressive stress at any stage during the deposition, and does not disclose that the compressive stress of the deposited nickel is maintained through 15 to 20 MTO. Moreover, Harbulak et al. does not disclose any of the metal salt stabilizers required by claim 1, nor do they place any importance on selecting metal sources comprising volatile anions to enhance not only the useful life of the electrolyte but to also affect the stress properties of the nickel layer such that the nickel layer has compressive stress through 14 to 22 MTO.

The Office's *prima facie* case of obviousness is therefore deficient for substantially the same reasons stated above. Briefly, the cited references do not disclose an electroless deposition method employing an electrolyte that has an extended useful life that further deposits a nickel layer having internal compressive stress throughout the useful life of the composition.

Moreover, applicants select nickel salts and metal stabilizers salts comprising volatile anions since such volatile anions enhance both the stability of the electrolyte and ensure that the deposited nickel layer is characterized by internal compressive stress and such compressive stress is a characteristic of the nickel layer for the entire extended throughput. Both cited references are entirely silent on the selection of bath compositions to thereby influence stress characteristics of the nickel layer.

Finally, the *prima facie* case of obviousness is effectively rebutted since applicants' method for depositing a nickel layer having internal compressive stresses for up to 22 MTO is an unexpected result that effectively rebuts the *prima facie* case of obviousness. See above and MPEP §2145. In this regard, applicants request that the Office refer to Part II.A.4 of this amendment and the attached Declaration of Inventor Franz-Josef Stark.

Claims 49, 52, 53, 59, 61, and 66 depend from claim 48 and are therefore patentable for the same reasons as claim 48 and by virtue of the additional requirements therein.

III. New Claims

Claims 67-69 depend from claim 48 and are therefore patentable for the same reasons as claim 48 and by virtue of the additional requirements therein.

CONCLUSION

In view of the foregoing, applicants respectfully request reconsideration and allowance of the pending claims.

Respectfully submitted,

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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application of Franz-Josef Stark et al. Art Unit 1792
Serial No. 10/678,601
Filed October 3, 2003
Confirmation No. 6149
For PROCESS AND ELECTROLYTES FOR DEPOSITION OF METAL LAYERS
Examiner Katherine A. Bareford

DECLARATION UNDER 37 C.F.R. §1.132

I, Franz-Josef Stark, declare and state as follows:

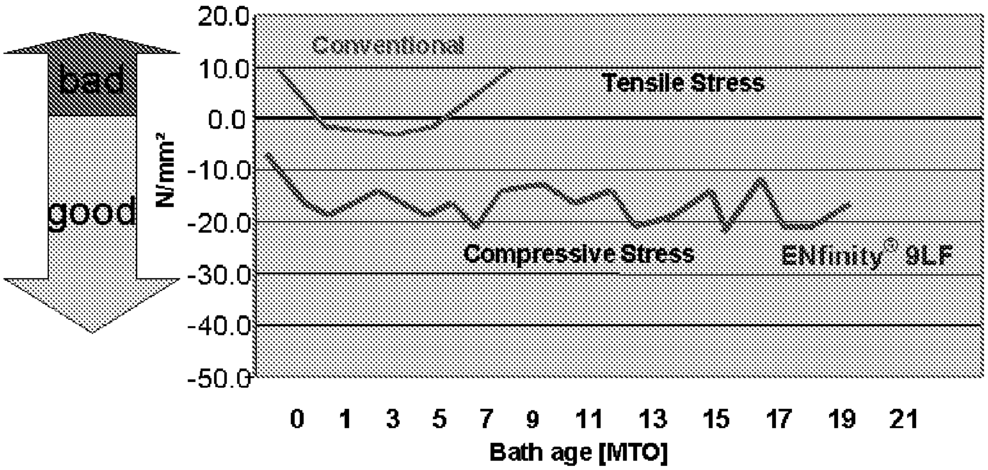
(1) I am co-inventor of the subject matter of the above-identified U.S. patent application, Serial No. 10/678,601, and a co-inventor of the subject matter of pending claims 48, 49, 52, 53, 59, 61, and 66-68.

(2) I have reviewed the Office Action issued November 20, 2007. I have reviewed all of the pending claims of this application, and the references cited by the U.S. PTO, including GB 1,243,134; Martyak et al. (U.S. 5,258,061); Zhong et al. (U.S. 6,106,927); and Harbulak et al. (U.S. 4,483,711).

(3) Not all electroless nickel plating compositions deposit nickel layers having internal compressive stresses through multiple metal turnovers. This is demonstrated by the two compositions I prepared as shown in the following table, labeled 1 and 2:

Composition	**1**	**2**
$NiSO_4 \cdot 6H_2O$ (g/L)	0	26.2
Nickel acetate·$4H_2O$ (g/L)	20.9	0
Sodium Hypophosphite·$1H_2O$ (g/L)	40	28
Lactic Acid (88%) (g/L)	45	21.6
Malic Acid (g/L)	0	14.1
Aminoacetic acid (g/L)	0	4
Disodium tetraborate·$10H_2O$ (g/L)	0	3.6
Sodium Acetate (g/L)	0	11
Malonic Acid (g/L)	0.25	0
Lead acetate (mg/L)	0.366	1.6
Cadmium acetate (mg/L)	0	1.9
Sodium Saccharine (g/L)	5	0
NaOH 33% solution (mL/L)	26.15	18.75

(4) Composition 1 is an electroless nickel deposition composition that falls within the scope of the claims. All metal sources (i.e., nickel ions, lead ions) contain volatile anions (acetate). Moreover, the complexing agent is lactic acid (2-hydroxypropanoic acid). Composition 2 is a conventional composition, which contains nickel sulfate hexahydrate, the preferred nickel source in the cited references and used most often in their Examples. The conventional composition also contains acetate and lactic acid. I deposited nickel layers from both compositions on substrates and periodically replenished the bath with maintenance solutions until the degree of reaction products (e.g., impurities, extraneous anions) rendered the composition unstable or adversely affected the quality of the deposit. The deposited nickel layers were measured for compressive stress through each metal turnover, with the results shown in the following graph:



Superior deposit characteristics

-Compressive Stress-

(5) The above graph shows that the conventional composition employing nickel sulfate hexahydrate is useful for about 8 MTO before it becomes unstable. The nickel layer deposited by the reduction of the initially added nickel ions has a tensile stress (indicated by the positive stress value in N/mm^2). After 1 MTO, the nickel layer has a slight compressive stress, but after 5 MTO, the deposited nickel layer is again characterized by tensile stress. The bath becomes unstable and plating stops after 8 MTO. The electroless nickel composition of the present invention, however, deposits a nickel layer having a compressive stress (negative stress values) initially and throughout the entire useful life of 20 MTO.

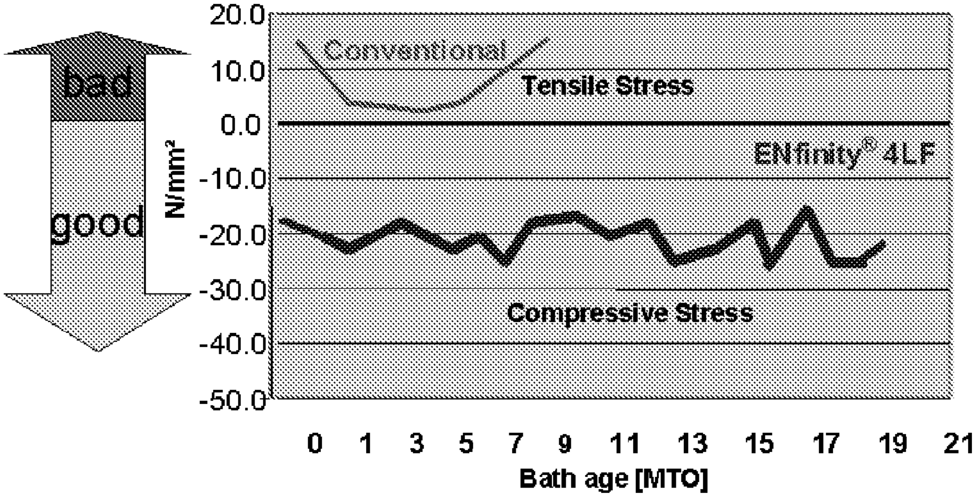
(6) The above effect is reproducible in "low phosphorus" electroless nickel compositions. This is demonstrated by the two compositions I prepared as shown in the following table, labeled 3 and 4:

Composition	**3**	**4***
Nickel Hydroxy Carbonate (g/L)	0	18-22
Nickel Acetate·4H_2O (g/L)	20.9	0
Sodium Hypophosphite·1H_2O (g/L)	50	10-13
Aminoacetic acid (g/L)	22.5	
Lactic acid 80% (mL/L)		?
Ammonia difluoride (g/L)		?
Ammonia 25% (g/L)	8.6	50
Bismuth acetate (mg/L)	3	
Lead Acetate (mg/L)		6.5-8.5

*Composition 4 is a commercial product marketed by a competitor. Although its general components are known, the exact concentrations of some components are not known.

(7) Composition 3 is an electroless nickel deposition composition that falls within the scope of the claims. All metal sources (i.e., nickel ions, bismuth ions) contain volatile anions (acetate). Composition 4 is a conventional composition, which uses nickel hydroxy carbonate as its nickel source. I deposited nickel layers from both compositions on substrates and periodically replenished the bath with maintenance solutions until the degree of impurities rendered the composition unstable or adversely affected the quality of the deposit. The deposited nickel layers were measured for compressive stress through each metal turnover, with the results shown in the following graph:

Unique compressive stress deposits for Low Phos



(8) The above graph shows that the conventional bath employing nickel sulfate hexahydrate is useful for about 9 MTO before it becomes unstable. Moreover, the conventional bath deposits a nickel layer that has a tensile stress (positive stress values) throughout the useful life of the composition. The electroless nickel composition of the present invention, however, deposits a nickel layer having a compressive stress (negative stress values) starting with the initial bath makeup through 20 MTO.

(9) The above effect is reproducible in high phosphorus electroless nickel compositions. This is demonstrated by the two compositions I prepared as shown in the following table, labeled 5 and 6:

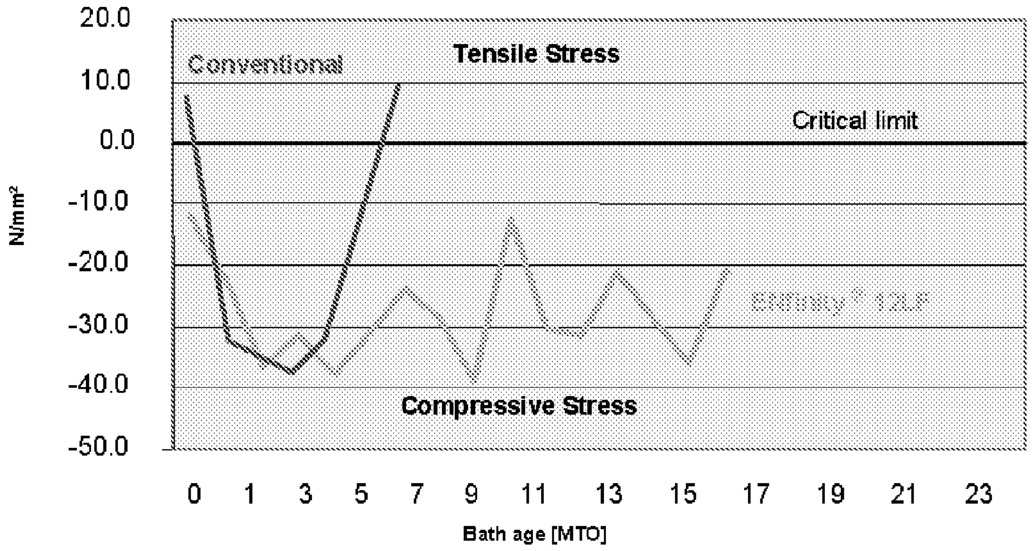
Composition	**5**	**6**
$NiSO_4 \cdot 6H_2O$ (g/L)	0	26.0
Nickel acetate$\cdot 4H_2O$ (g/L)	29.3	0
Lactic acid 88% (g/L)	51.5	40.0
Sodium Hypophosphite$\cdot 1H_2O$ (g/L)	40	20.3
Malonic acid (g/L)	0.25	0

Composition	**5**	**6**
Malic Acid (g/L)	0	11.9
Antimony acetate (mg/L)	13	
Lead Nitrate (mg/L)		0.58
Sodium Saccharine (g/L)	5	0
NaOH 33% solution (mL/L)	32	46.40

(10) Composition 5 is an electroless nickel deposition composition that falls within the scope of the claims. All metal sources (i.e., nickel ions, lead ions) contain volatile anions (acetate). Moreover, the complexing agent is lactic acid (2-hydroxypropanoic acid). Composition 6 is a conventional composition, which contains nickel sulfate hexahydrate. The conventional composition also contains acetate and lactic acid. I deposited nickel layers from both compositions on substrates and periodically replenished the bath with maintenance solutions until the degree of impurities rendered the composition unstable or adversely affected the quality of the deposit. The deposited nickel layers were measured for compressive stress through each

metal turnover, with the results shown in the following graph:

Change of Internal Stress in High Phos EN Deposit



(11) The above graph shows that the conventional bath employing nickel sulfate hexahydrate is useful for about 6 MTO before it becomes unstable. The nickel layer deposited by the reduction of the initially added nickel ions has a tensile stress (positive stress values). At 1 MTO, the nickel layer has strong compressive stress, but after 5 MTO, the deposited nickel layer is again characterized by tensile stress. The bath becomes unstable and plating stops after 6 MTO. The electroless nickel composition of the present invention, however, deposits a nickel layer having a compressive stress (negative stress values) through about 16 MTO.

(12) In view of the foregoing and in view of the reproducibility of these results, I conclude that the use of volatile anions in the metal ion sources is a critical feature of the electroless nickel plating method of my application,

since that use of volatile anions increases the useful life of the electroless nickel composition and further causes a nickel layer having internal compressive stress to be deposited throughout the composition's useful life.

(13) I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further, that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S.C. 1001, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Franz-Josef Stark

05-09-2008
Date